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ANALYSES OF HIGH TEMPERATURE RECOVERY AND COMPRESSION SET TESTS

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SUPPLEMENT I TO THE FINAL REPORT "PREFORMED ELASTOMERIC JOINT SEALERS FOR BRIDGES ..... PHASE II

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This report is a supplement to a final report entitled "Preformed Elastomeric Joint Sealers for Bridges.....Phase II"

16. Abstract

This supplement describes an attempt to develop tests that would rectify certain inadequacies of the current New Jersey Department of Transportation's specification for preformed elastomeric compression joint sealers. This specification in its current form was found to be unable to differentiate between sealers having good and poor functional durability. From consultation with rubber manufacturers it was apparent that the specification needed a mechanism for identifying basic quality deficiencies in the sealer material related to inconsistent manufacturing processes. Two possible quality indicator tests for sealer material were considered - the High Temperature Recovery Test and the Compression Set Test (ASTM D-395-69 Method B). After a joint evaluation effort with the DuPont deNemours Company, the Compression Set Test was selected for possible use by the New Jersey Department of Transportation. The ability to isolate, to some degree, inadequate sealer material by the Compression Set Tests was established. For the New Jersey Department of Transportation's laboratory evaluations an upper acceptance limit on Compression Set of 32% based on a sample of not less than 20 pellets is recommended. Unfortunately, use of this test limit by other agencies for sealer acceptance cannot be proposed at this time.

Excessive between-laboratory variability, as found in our studies, makes it difficult for the acceptance limit developed for the New Jersey Department

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Abstract (Continued)

of Transportation to be used by others.

The researchers conclude that the Department's use of compression set requirements will at least serve to tighten up the sealer material specification and make it more difficult for poor or marginal sealers to get into service. It is thus evident that sealer manufacturers still hold the key to the consumer getting a uniformly durable product and they must take the lead in assuring that this is achieved.

ACKNOWLEDGEMENT

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The Federal Highway Administration has provided the financial means for performance of this research and patiently awaited results. The FHWA's continued support and cooperation is gratefully acknowledged.

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The assistance and the cooperation of the Bureau of Quality Control, in particular that of Mr. Juno Vanelli, is most appreciatively acknowledged.

The interest, help and cooperation of E. I. DuPont DeNemours and Company organization and in particular Mr. Robert H. Barton also contributed significantly to the success of Phase II of this research.

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1. Introduction and Background

As stated in the main report[1] ("Preformed Elastomeric Joint Sealers for Bridges....Phase II") which this document supplements, the purpose of Phase II of the research effort was to develop realistic material specifications closely related to the actual field performance requirements of a preformed elastomeric compression sealer. In pursuit of that goal, a special test program was undertaken to determine the adequacy of current sealer specifications. This program initially involved an attempt to correlate specification conformance with sealer performance in a simulated bridge joint environment. Subsequent efforts concentrated on developing other specification criteria that could serve as better predictors of the durability characteristic. These latter activities will be the subject of this report. For sequence of events to follow see the flow chart in Figure 1.

The most essential factor to keep in mind is that the Phase II research effort presents a very unique case. To date, it is the only study of preformed sealers for which basic sealer properties have been related to durability life limits through CONTROLLED PERFORMANCE tests under TRUE FIELD EXPOSURE conditions. This is accomplished with the aid of Model Joint Machines (outdoor joint simulators) as shown in Figure 2 and described in the main report.

Compression sealers representative of sizes and types (manufacturers) predominantly used by the Department were selected and tested in accordance with the Department's normal acceptance specification. Sealer specimens found acceptable were then inserted

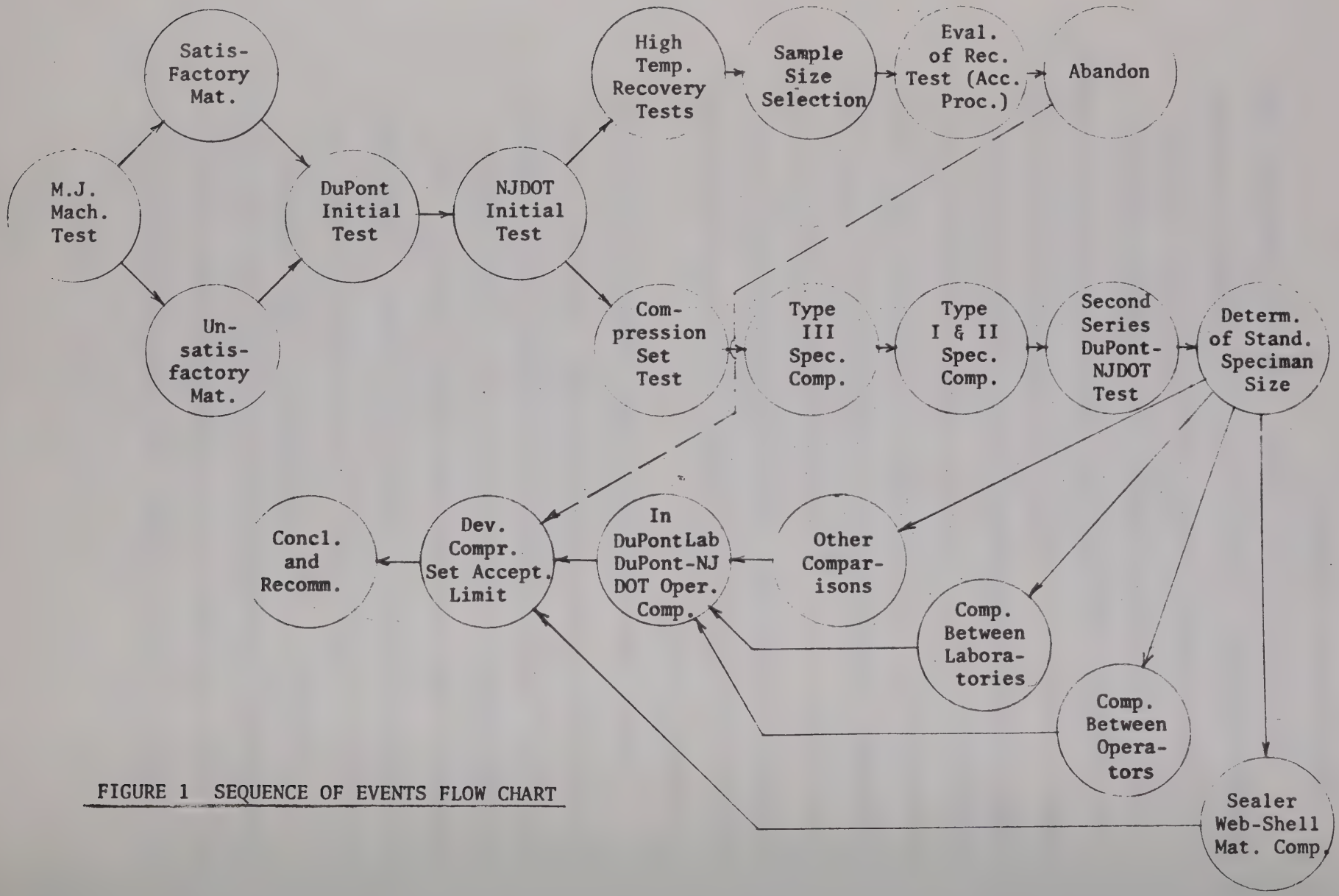

FIGURE 1 SEQUENCE OF EVENTS FLOW CHART

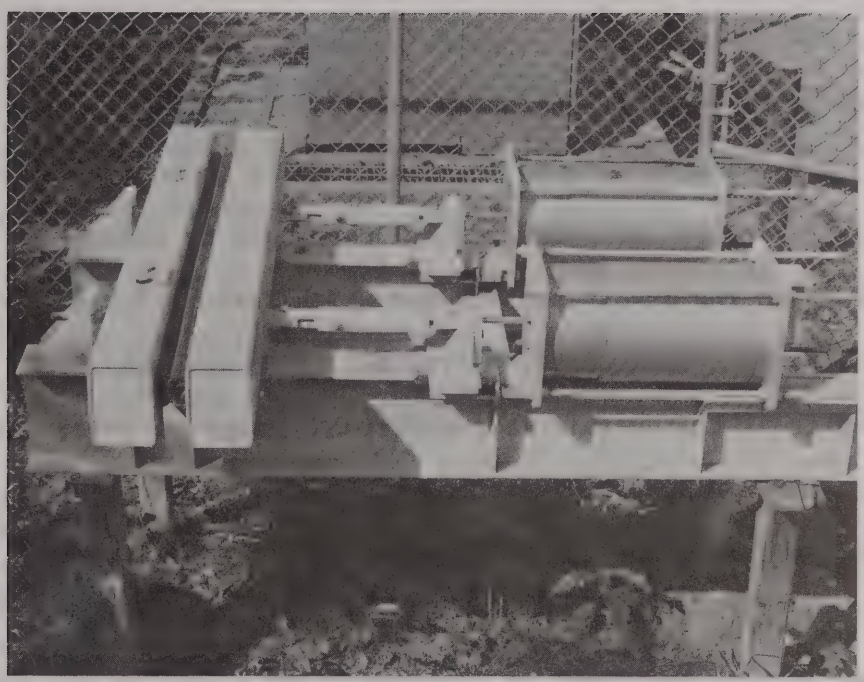

FIGURE 2 - NON-SKEW MODEL JOINT MACHINE.

in the Model Joint Machines for environmental simulation. Periodically, certain non-destructive tests were made on the sealer to gauge property changes with time. Finally, when sealer failure occurred or the time of its future failure was sufficiently predictable each sealer was retested to determine time related changes for all acceptance specification properties.

For this research a sealer's failure point was defined as that time in a sealer's life at which the sealer exerts a pressure of only 0.5 to 1.0 psi when allowed to expand to its maximum field design width. This level of pressure has been found by experience in New Jersey to be the lowest pressure at which sealer leakage and solids intrusion can be reasonably prevented. Lots 150, 153, 402, 406 and 474 represent the overall life expectancy range of these more desirable sealers and were selected for subsequent evaluative comparisons to the short life sealer.

Attempts to correlate durability with levels of conformance to the New Jersey Department of Transportation's sealer specification proved fruitless as none of the normal specification tests apparently yielded results relating significantly to observed sealer life. This determination combined with the fact that all compliant sealers exhibited a one-in-four occurrence of poor durability indicated the need for additional investigation.

It is evident that the life expectancy of the finished sealer is dependent upon the manufacturing variables involved in the creation of that product. Unfortunately, an evaluation of the manufacture of sealers is not within the scope of this project. Also, since the commercial producers insist on shrouding their particular manufacturing techniques and details in secrecy, further investigation

along these lines is hindered, if not impossible. On the other hand, refraining from a pursuit of this problem would leave the user practically without any means for determing some obviously inferior products.

The only available course in this matter was then to turn to industry. Fortunately from the start of this research, some 10 years ago (see Reference 1) New Jersey researchers enjoyed the support, advice, and respect of many members of the DuPont deNemours organisation. Therefore, in order to pursue the sealer performance problem further, portions of leftover sealer specimens from certain of the original Model Joint Machine test specimens were sent to DuPont for various chemical and mechanical analyses deemed necessary by them. Leftovers of Lots 6, 18, 25, and 71 henceforth called "unsatisfactory" material and of Lots 150, 153, 402, and 406 subsequently called "satisfactory" material were used in the analyses. The results of the DuPont evaluative effort suggested some additional quality indicator tests which could be expected to identify, to some degree, the unsatisfactory material. This report provides the details and data on the joint development of the two tests DuPont proposed for addition to the overall acceptance specification for preformed elastomeric compression sealers.

Out of necessity the work described herein ends New Jersey's effort at stepping into the manufacturer's field to improve the sadly lacking and badly needed quality controls for production of elastomeric compression sealers.

2. DuPont's Initial Test Series

As a result of DuPont laboratory analyses of the "satisfactory" and "unsatisfactory" materials, two possible quality indicator tests were suggested for consideration - a compression set test and a high temperature recovery test.

The use of the compression set test in the ASTM and the Department's sealer specifications had been considered in the early 1960's[1] when the specification was first developed. However, in discussions with industry it was learned that the preparation of test specimens from finished bridge seals was extremely difficult with the consequence that there would be a high likelihood of error in test results. The high temperature recovery test was already a part of the Department's specification but DuPont was proposing the testing time be increased from the standard 70 hours.

In the process of the DuPont investigation, in the winter of 1974, the compression set test was performed by DuPont on 0.51 $\pm$ 0.01 inch diameter by 0.50 $\pm$ 0.02 inch thick plied pellets of the unsatisfactory and satisfactory sealer materials. All testing was done in accordance with the ASTM D-395-69 Method B specification[2]. The specimens were aged 70 hours at 212°F with the results as indicated in Table 1 being obtained.

Without statistical analyses the difference in results between unsatisfactory and satisfactory lots appeared as being sufficiently distinct.

TABLE 1 COMPRESSION SET DATA FOR

DUPONT'S INITIAL TEST SERIES

Lot No.	Compression Set (%)	
	Inner Material (Webs)	Outer Material (Outside Skin)
Unsatisfactory		
6	43.6	53.6
18	42.8	48.3
25	38.5	48.6
71	39.1	45.2
Satisfactory		
150	35.2	26.9
153	26.7	27.3
402	34.1	35.3
406	31.3	31.3

DuPont's efforts then turned to a trial of High Temperature Recovery test using a modified ASTM-395-69 test method. However, the tests did not look so decisive. This test was performed on 6 inch long sealer specimens under 50% compression and aged at 212°F with the results being those given in Table 2.

TABLE 2 HIGH TEMPERATURE RECOVERY DATA

FOR DUPONT'S INITIAL TEST SERIES

Lot No.	Original Width (In)	% Recovery after 70 hrs.	% Recovery after 140 hrs.	% Recovery after 210 hrs.
Unsatisfactory				
6	2.719	87.2	80.4	76.4
18	2.730	91.4	85.8	80.2
25	2.743	86.9	79.7	74.4
71	2.744	88.8	81.6	75.6
Satisfactory				
150	4.230	91.6	87.2	84.2
153	2.670	92.4	87.4	83.2
402	4.193	91.2	85.1	81.0
406	2.450	90.5	85.9	81.6

On the basis of the foregoing test data the suggested revisions to the Department's preformed elastomeric compression bridge joint sealers specifications were as follows:

1. Add a compression set test with a 35% maximum set being permitted after aging 70 hours at 212°F.

2. Add a high temperature recovery test having an 80% minimum recovery limit after 210 hours of aging at 212°F.

While reinstatement of the compression set test was tentatively suggested without apparent general dissension, opinions offered concerning the high temperature recovery tests were divided. Some suggested discarding it because of the excessively long testing time and the indecisiveness of the results. However, New Jersey was reluctant to entertain that suggestion until DuPont results were duplicated in New Jersey and a more substantial investigation was conducted. In the past, as stated previously in this report, the compression set test was abandoned because when performed on samples obtained from finished sealers, the results can be very much clouded by error. On the other hand a high temperature recovery test that lasts 210 hours is rather impractical.

The concern about these recommendations and the need to establish New Jersey Department of Transportation's test procedures led to the work described hereinafter and called "NJDOT" testing efforts.

3. New Jersey Department of Transportation's Initial Testing Efforts

3.1 General

Primarily to achieve the best possible repeatability between laboratories, a qualified individual from the New Jersey Department of Transportation visited DuPont's laboratory with their consent. After his return, attempts were made at the compression set and high temperature recovery tests with disappointing results, i.e. no agreement between the two laboratories' test results could be

established. It must be pointed out that both agencies followed the ASTM D-395-69 Method B test procedure[2] closely. However, the recovery test procedure was a modification of the cited ASTM procedure which is described in the New Jersey Department of Transportation specification[3].

To determine causes of discrepancies in results between laboratories, a thorough check of procedures in both laboratories followed using steps suggested by DuPont. In the NJDOT laboratory it was established that ASTM procedures were followed meticulously, workmanship appeared to be excellent and the equipment functioned satisfactorily. The oven, the most critical piece of equipment, was checked electronically, the results indicating that the temperature throughout the oven was controlled within $\pm$ 2°F of the desired value.

However, in the compression set test, DuPont modified the ASTM specification slightly by the use of 0.51 $\pm$ 0.01 inch diameter by 0.50 $\pm$ 0.02 inch thick plied pellets while the NJDOT followed the ASTM specifications exactly. In essence, the two agencies did not test the same type of specimen. The effect of this modification caused some of the aforementioned discrepancy, leading to another testing program that started in the spring of 1974, the results of which have been analyzed statistically to determine the degree of variability between the two laboratories. This second testing program which included both the high temperature recovery and compression set tests will be described in detail in the remainder of this section of the report.

3.2 High Temperature Recovery Test

3.2.1 Selection of Sample Size

Since only very little Model Joint Machine tested sealer material was left for NJDOT testing, an attempt was made to check whether a three (3) inch sample could be used instead of the customary 6 inch length of specimen without detrimentally affecting the test results. Accordingly, the first series of tests was designed to compare the results obtained with the two different specimen sizes.

The test was performed, as suggested by DuPont, under 50% compression at 212°F for a period of 210 hours. Eleven (11) lots of extra material (other than Model Joint Machine tested material) representing a wide range of quality were used and the results are presented in Table 3. Although there seems to be no difference in the mean recovery values obtained with these two specimen sizes, there does appear to be a significant increase in the variability (standard deviation) of the tests made with the smaller (3") specimen. Since it is a fundamental principle of statistics that the means of smaller samples exhibit greater variability, this is interpreted to result from the fact that a 3" specimen is a smaller sample of the material than a 6" specimen and it indicates that there must be some degree of variability within the material itself. As a result, it would ordinarily be desirable to select the larger specimen. However, because a very limited amount of material was available for the next series of tests, it was decided to select the 3" size so that there would be a sufficient number of samples to provide a reasonable estimate of the precision of the process.

TABLE 3 RECOVERY TESTS - 6 INCH VS 3 INCH SAMPLES

Lot Number	6" Samples			3" Samples			Difference of Means
	Number of Tests	Mean Recovery	Standard Deviation	Number of Tests	Mean Recovery	Standard Deviation	
088368	1	74.7	--	1	82.2	--	-7.5
WA200	6	77.9	3.03	6	74.4	3.31	3.5
70	1	70.2	--	1	67.2	--	3.0
52	2	47.7	7.92	2	56.1	0.57	-8.4
075966	1	85.4	--	1	86.1	--	-0.7
WD200	5	61.1	1.50	5	65.5	8.37	-4.4
086443	1	87.7	--	1	86.2	--	1.5
093421	2	87.0	1.06	2	85.2	1.84	1.8
093422	2	81.9	0.57	2	81.1	0.85	0.8
65	1	79.2	--	1	79.1	--	0.1
42000	6	79.0	3.39	6	76.2	2.38	2.8
Weighted Averages:		74.19	3.22*		74.17	4.65*	0.02

*Note: Pooled standard deviations are significantly different at the 90% confidence level but not at the 95% level as indicated below:

$$F_{calc} = \frac{4.65^2}{3.22^2} = 2.09, \quad F_{17,17,0.95} = 2.28, \quad F_{17,17,0.90} = 1.89$$

3.2.2 Evaluation of Recovery Tests as an Acceptance Procedure

The second series of tests was intended to determine an appropriate acceptance procedure to distinquish between satisfactory and unsatisfactory material. As before, the test was performed under 50% compression at 212°F for a period of 210 hours.

In order to judge the suitability of this test for predicting the field performance of a sealer, tests were run on material from 8 lots whose quality had been determined by their performance in the Model Joint Machines. The limited amount of material permitted only 2 tests per lot and the results of these are summarized in Table 4. Although the overall means of the satisfactory and unsatisfactory material seem to be fairly well separated (81 and 75, respectively), a problem is immediately apparent when the individual lot means are studied. Remembering that higher recovery values are more desirable, one can see that one of the known "unsatisfactory" materials (Lot 18 with a mean recovery of 79.8) scored higher than one of the known "satisfactory" materials (Lot 406 with a mean of 77.3). This suggests that the recovery test is not always a reliable indicator of the expected field performance of a sealer and that, if it is used to judge the acceptability of this material, a number of incorrect decisions may be made.

It is acknowledged that more data would have been desirable but due to the limited quantities of material available, this was not possible. If it had been possible to increase the number of tests for each lot in Table 4, a more clear-cut separation of "satisfactory" and "unsatisfactory" materials might have been found.

TABLE 4 RECOVERY TESTS OF "SATISFACTORY"

AND "UNSATISFACTORY" MATERIAL

Lot Number	Quality	Number of Tests	Mean Recovery	Standard Deviation
150	Satisfactory	2	84.0	0.00
153	Satisfactory	2	82.9	0.28
402	Satisfactory	2	79.8	0.21
406	Satisfactory	2	77.3*	0.42
6	Unsatisfactory	2	76.4	0.49
18	Unsatisfactory	2	79.8**	0.35
25	Unsatisfactory	2	77.2	0.28
71	Unsatisfactory	2	66.5	0.85

*Worst "Satisfactory" Lot
**Best "Unsatisfactory" Lot

Note: Difference between these two particular lots is significant at 95% confidence based on the following statistics:

Calculated t = 6.46

Table $t_{2,0.95}$ = 4.30

	Overall Mean Recovery	Pooled Standard Deviation
Satisfactory:	81.0	0.27
Unsatisfactory:	75.0	0.54

However, it is also possible that the opposite effect might have occurred. Based on the available information it is not deemed feasible and/or practical to proceed with the development of an acceptance procedure utilizing 210 hour high temperature recovery test either as replacement for the Department's present 70 hour high temperature recovery test or as an addition to the present material qualification tests.

3.3 Compression Set Test

3.3.1 Background and Objectives

The primary objective of the Department's trials of the compression set test was to set realistic material acceptance limits based on these tests and to establish the validity of said limits. In initiating this work one critical question to be answered was: which type of specimen would provide the most accurate evaluation of the material being tested?

The testing procedure to be used for determining a sealer's compression set characteristics, as already stated, is the standard ASTM D-395-69 Method B[2] Test for Compression Set of Vulcanized Rubber, modified by utilization of Type III plied pellets as hereinafter described. The standard method requires testing to be performed on a solid or plied piece of vulcanized rubber. These specimens must be either of certain dimension, identified as Types I and II or of any convenient size such as that introduced by DuPont in their testing program (called Type III in this report). The shell and web thicknesses of preformed sealers are relatively thin necessitating a plying of pellets to achieve the required sample thickness.

The specific dimensions of the different sample types that were considered in the Department's testing efforts are given below:

	Nominal Dimensions	
Types	Diameter (in)	Thickness (in)
I	1.14 ± 0.02	0.50 ± 0.02
II	0.51 ± 0.01	0.24 ± 0.01
III	0.51 ± 0.01	0.50 ± 0.02

3.3.2 Type III Specimen Tests

As already indicated, NJDOT followed ASTM specifications using, in essence, Type I specimens while DuPont results were obtained with what was described in the previous tabulation as Type III specimens. Since the relationship between thusly established compression set data was not known, this initial effort could not be compared. However, because of the possible advantages associated with specimens of a smaller size, especially where small sealers are involved, it was decided to carry out the second series using the Type III specimens.

The results of the NJDOT tests on Type III plied pellets were obtained and compared to the aforementioned initial DuPont tests. A regression of the results of the two agencies' tests is shown in Figure 3.

The analysis in Figure 3 shows a highly significant relationship between the results obtained by the two laboratories with a definite tendency for the DuPont tests to produce consistently higher values (indicating poorer performance of the material). It also became evident that the Type III specimens, when compared to the

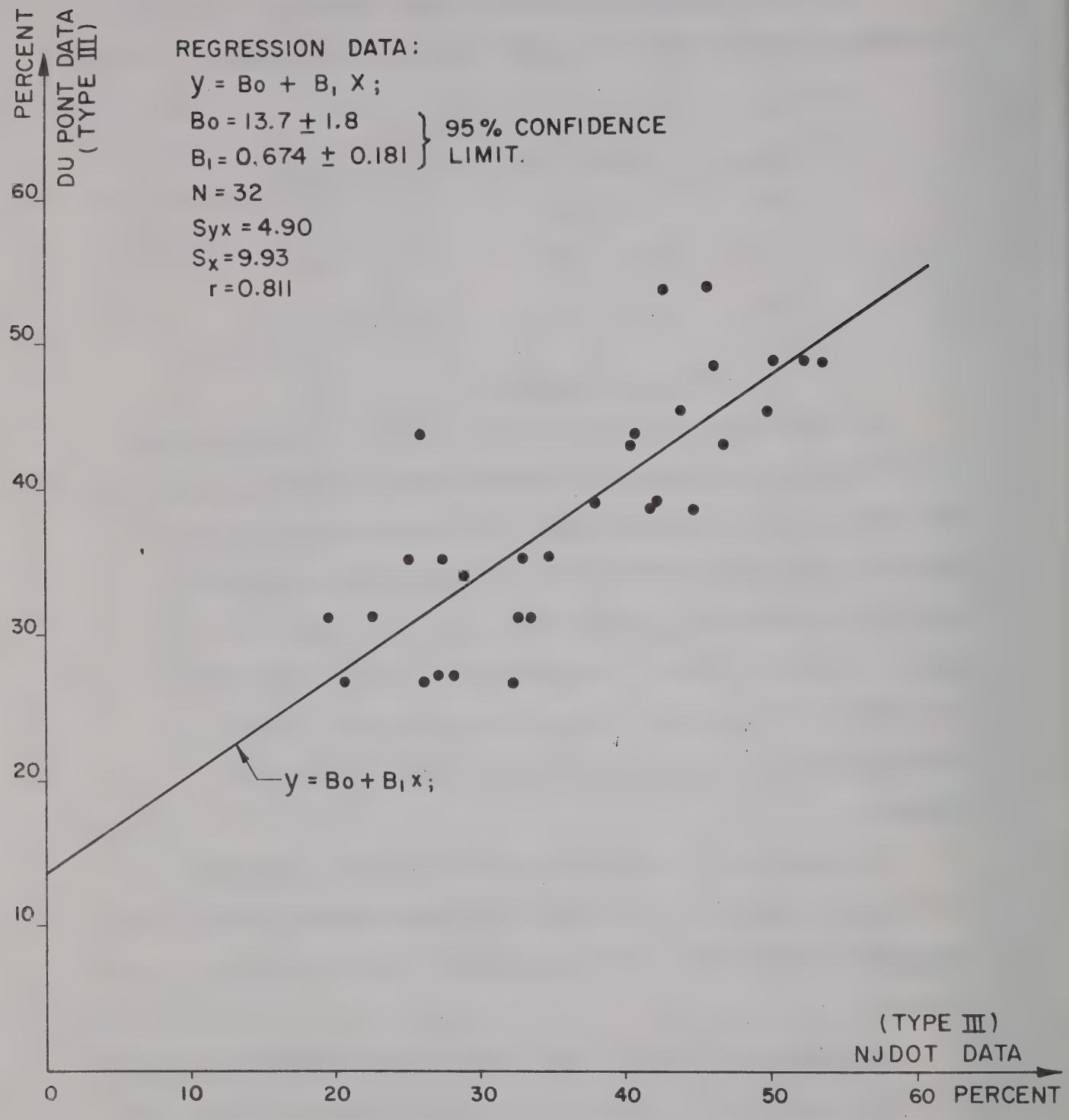

FIGURE #3 REGRESSION ANALYSIS OF NEW JERSEY AND DUPONT COMPRESSION SET DATA FOR LOTS 6,18,25,71,150,153,402,& 406.

Type I specimens (data not shown), produced more consistent results thus making the analysis of the effect of specimen sizing a logical item to be pursued in greater detail. This was particularly evident since the dimensions of some sealer sizes frequently made it difficult or even impossible to cut out Type I (1.14 inch diameter) sample disks.

3.3.3 Type I and II Specimen Comparison

In order to pursue the matter of specimen size further, it was decided to perform some comparative tests on Type I and Type II specimens. Although only limited amounts of extra random material could be provided for performance of compression set tests involving Type I and II specimens, the results appear to be rather interesting as can be seen below:

Compression Set Test of Lot #098022

Type	Outer Shell			Inner Webbing		
	Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)	Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)
I	12	22.2	1.97	12	24.8	3.24
II	15	24.4	3.21	16	25.2	4.29

It appears that the Type I specimens produced less variable (i.e., more precise) compression set results than the Type II specimens. One possible explanation for this is the quantity of individual disks plied together to form the specimens. Normally, of course, slippage between layers could be expected to produce erroneous test results. However, the Type I specimens, because they were twice as thick, contained twice as many layers as the Type II specimens. And since

each layer can be thought of as an individual sample from an approximately random location within the material, a Type I specimen can be considered as being equivalent to a random sample which is twice as large. Therefore, any component of variance related to within material variability is automatically divided by two. Since almost any material has a substantial degree of variability throughout its mass, it was felt that the thicker specimens with a greater number of layers would probably continue to produce better results.

The results of these tests were discussed with DuPont and it was agreed that a joint testing effort should be initiated to isolate the optimum specimen size, and to gain more data on test precision. Since Type I specimens were rather impractical and Type III appeared to offer relatively consistent results, it was decided to perform the next set of tests on Type II and III specimens in both DuPont and NJDOT laboratories.

4. Second DuPont-New Jersey Department of Transportation Test Series

4.1 General

The primary interest in the second testing program was to determine which size of specimen would produce the more precise (repeatable) results in order to standardize this type specimen for future tests.

For this series of tests, DuPont produced four special lots of slabs of a variety of neoprene compositions. These 6x6x1/8 (inches) lots were coded - Number 73 815 - 68, 69, 70 and 71 representing two different curing systems and material formulations as shown in the

table below:

Lot	Composition	Cure
68	Normal	#1
69	Degraded	#1
70	Normal	#2
71	Degraded	#2

According to DuPont the degradation of the compound was produced basically by addition of clay. The difference in cures, however, has been created by addition of NA-22 (Lots 68 and 69) in Cure #1 and of Thiate E and Epon 828 (Lots 70 and 71) in Cure #2.

4.2 Determination of Standard Specimen Size

In the spring of 1975 approximately 40 compression set tests each were run on material corresponding to the 4 formulation-cure combinations listed above by both the DuPont laboratory and the NJDOT laboratory. The statistical results of these tests are summarized in Table 5. Using the data of Table 5, it is possible to compare the standard deviations (S) for the two specimen Types II and III over a range of four different quality levels (Lots 68-71). If one of the specimen sizes produces consistently lower standard deviations, the size can be judged to be preferable. The results of this comparison are presented in Table 6.

It can be seen from the data in Table 6 that there is a consistently significant improvement in precision obtained with the Type III specimen size. Therefore, this specimen size (Type III) appears to be the best choice for adoption as a standard for the

TABLE 5 SUMMARY OF STATISTICAL DATA

FOR COMPRESSION SET TESTS ON

6x6x1/8 (INCHES) SLABS

Laboratory	Lots	Specimen Types					
		II			III		
		Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)	Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)
NJDOT	68	44	28.67	3.22	44	33.18	3.87
	69	48	33.36	2.99	47	36.03	1.21
	70	44	22.10	1.52	44	23.15	0.94
	71	48	23.14	2.17	49	26.08	1.65
DuPont	68	40	43.21	3.41	40	40.87	1.69
	69	40	48.42	1.97	40	46.77	1.25
	70	40	33.49	2.14	35	32.40	1.10
	71	40	37.31	1.87	30	37.28	0.99

TABLE 6 COMPARISON OF SPECIMEN STANDARD DEVIATIONS

Laboratory	Lot	Standard Deviation		Calc.* F	Critical F (95%)	Significant Difference
		Type II	Type III			
NJDOT	68	3.22	3.87	1.44	1.66	No
	69	2.99	1.21	6.11	1.61	Yes
	70	1.52	0.94	2.61	1.66	Yes
	71	2.17	1.65	1.73	1.61	Yes
DuPont	68	3.41	1.69	4.07	1.69	Yes
	69	1.97	1.25	2.48	1.69	Yes
	70	2.14	1.10	3.78	1.74	Yes
	71	1.87	0.99	3.57	1.79	Yes

*$F = S_1^2/S_2^2$ is the statistic used to test if two sample standard deviations, S_1 and S_2, are significantly different from one another.

compression set tests. All further analysis presented in this report is based on results from Type III specimens.

4.3 Other Comparisons

The initial series of DuPont tests did not establish causes for poor performance of the "unsatisfactory" sealers. Thus, when planning the second series it was purposely decided to use a range of cures or formulations of material. It was hoped that the test results would isolate one of these factors to be a possible key to the sealer performance difference detected in the Model Joint Machine evaluations.

After the choice of Type III specimen size was made, several further comparisons became possible using the data of Table 5. For example the two types of curing were also compared, as shown in Table 7.

TABLE 7 COMPARISON OF TWO METHODS OF CURING

Laboratory	Lot and Mean Compression Set				Change in Compression Set Apparently Due to Cure #2
	Lot	Cure #1	Lot	Cure #2	
NJDOT	68	33.18	70	23.15	-10.03
	69	36.03	71	26.08	- 9.95
DuPont	68	40.87	70	32.40	- 8.47
	69	46.77	71	37.28	- 9.49

Since a difference of about one unit in the compression set means would be statistically significant, the differences shown in Table 7 are obviously highly significant and suggest that the "Cure #2" appears to be extremely beneficial in reducing the degree of compression set.

Another comparison that could be made was between the "normal" composition and the formulation "degraded" by the addition of clay.

Here we expected the degraded material to have a higher compression set and the results in Table 8 confirm that this is the case. Again only Type III specimens results have been used.

TABLE 8 COMPARISON OF TWO FORMULATIONS OF MATERIAL

Laboratory	Lot and Mean Compression Set				Change in Compression Set Apparently Due to Addition of Clay
	Lot	Straight	Lot	Degraded	
NJDOT	68	33.18	69	36.03	+2.85
	70	23.15	71	26.08	+2.93
DuPont	68	40.87	69	46.77	+5.90
	70	32.40	71	37.28	+4.88

4.4 Comparison Between Laboratories

Another very important comparison that can be made using the data in Table 5 concerns the differences in the results obtained by the two laboratories. Again, only Type III specimen data of Table 5 is used and the results of this comparison are shown in Table 9 Since both laboratories performed the same test on samples of the same material using similar equipment, it was thought that they would obtain very nearly the same results. However, as can be seen in Table 9, the values obtained by DuPont are consistently about 10 units higher than the values obtained by the New Jersey laboratory. The reasons for this are not known since no fault was apparent with either the procedures or the equipment. Furthermore, since the material used in this test was typical sealer material, the same large variance between laboratories could also be expected to occur with tests on actual sealers. The significance of this finding is that, based on this information, it appears impossible to determine an acceptance limit which will be standard for all laboratories. It appears that

each laboratory would have to determine its own appropriate acceptance limit based on tests of known "satisfactory" and "unsatisfactory" material, i.e., the kind of material that was available for these tests.

TABLE 9 COMPARISON BETWEEN LABORATORIES

Mean Compression Set			Between-Laboratory Difference
Lot	NJDOT	DuPont	(DuPont Mean - NJDOT Mean)
68	33.18	40.87	7.69
69	36.03	46.77	10.74
70	23.15	32.40	9.25
71	26.08	37.28	11.20

5. Comparison Between Operators in the NJDOT Laboratory

The results of the intra-laboratory comparison led to consternation concerning reliability of said tests within a laboratory. All of the NJDOT tests considered thus far were performed practically by the same operator because he was a trained technician and because a different operator would add another variable. This particular operator was considered to be very good and the question was raised as to whether he could be considered typical of all operators that might be involved with such tests in the future. Of course, DuPont operators most probably are also well trained and therefore should be good. Thusly, one could not escape the logical question as to why there was such a difference in test results between the two laboratories.

To answer this question, a second operator was trained and then a series of tests were performed with both NJDOT operators

receiving samples of the same sealer specimens.

The results of these tests are listed in Table 10. In 7 out of the 8 tests, the new operator had a higher standard deviation (i.e., was less precise) although only 3 of these 7 were actually significant at the 95% confidence level. This result was not unexpected because of the relatively short period of training the new operator had received and it was felt that, in time, he would be very nearly equal to the experienced operator.

In addition to the relative precision of the operators, there was also concern that there might be a bias in their mean results similar to that found between the DuPont and NJDOT laboratories (Table 9). Although there was a significant difference in the means in 5 out of the 8 cases, the differences were almost evenly divided in direction. In other words, the new operator obtained lower values in 3 out of the 5, and had higher values for the remaining 2. Thus there appears to be no consistent bias between the two operators. The 5 significant differences must then be attributed to material differences or other causes although the tests had been randomized in an attempt to avoid this.

Although the new operator's performance did not quite equal that of the experienced operator, it was reasonably close. We concluded from this that the results of the numerous tests made in this study can be considered typical for a trained, experienced operator.

6. Comparison between DuPont and NJDOT Operators Performed for the Compression Set Test in the DuPont Laboratory

To pursue the resolution of the problem of a consistent difference (10 units) of test results between laboratories, another

TABLE 10 COMPARISON OF OPERATORS

Material	Experienced Operator			New Operator			Means Signif. Diff. at 95%	Std. Devs. Signif. Diff. at 95%
	Number of Samples	Mean Compression Set	Standard Deviation	Number of Samples	Mean Compression Set	Standard Deviation		
A1-Outer	35	31.90	2.18	22	28.77	2.68	Yes	No
A1-Inner	31	35.22	6.16*	28	33.07	2.85	No	Yes
A2-Outer	38	29.04	2.26	17	35.68	2.70	Yes	No
A2-Inner	25	37.95	2.22	23	38.99	3.46	No	Yes
B1-Outer	32	33.38	1.59	28	35.20	2.68	Yes	Yes
B1-Inner	25	35.77	2.82	37	34.70	3.64	No	No
B2-Outer	28	32.25	2.34	35	27.69	3.78	Yes	Yes
B2-Inner	22	35.43	2.21	40	32.02	2.52	Yes	No

*Although no obvious error in the data could be found, this value is felt to be unrealistically large.

series of tests were performed during the period of July 27 to August 2, 1976. As before, the NJDOT operator who performed previous tests and his supervisor and the DuPont operator also under appropriate supervision performed compression set tests in DuPont's Chestnut Run Laboratories near Wilmington, Delaware. DuPont equipment and identical elastomeric rubber slabs were used. It was hoped that these tests performed in such controlled conditions would make it possible to isolate the cause of said consistent differences experienced in the previous tests.

6.1 Procedure

The test was performed in accordance with ASTM D-395-69 Method B using Type III (0.51 $\pm$ 0.01 inch diameter by 0.50 $\pm$ 0.02 inch thick) specimens. The original intent was to use at least 30 specimens for each operator and each quality of material. However, Type I specimens had been inadvertently prepared thusly reducing the number of the same type of specimens. Therefore, to keep at least as many specimens as possible for each operator the Type I specimens were also used.

Two rubber compounds were prepared at the Chestnut Run Laboratories (a "normal" material, Lot 68 and a "degraded" material, Lot 69) in three sheets approximately 6x6x0.125 inch in thickness. Pellets were cut in advance by DuPont operators by a drill press to the specified diameters. The distribution of the material was performed by randomly compiling the specimens by each operator from the pellets stockpile.

Both operators used the following procedure:

1. The pellets were each gaged individually with DuPont's

dial micrometer, although originally this step was not contemplated.

2. The pellets were then plied together and the composite thickness was gaged, again using DuPont's dial micrometer.
3. The compression set test was completed in accordance with the ASTM standards with additional constraints as follows:
    - 3.1 Only 0.375 inch spacers were used.
    - 3.2 Only DuPont equipment was used, except for NJDOT plates which were used in addition to DuPont jigs.
    - 3.3 As many specimens as practically possible were clamped in the same set of platens.
    - 3.4 As far as possible, the jigs of each operator were located side by side in the oven.
    - 3.5 In the test, as far as possible, the steps were performed by both operators in unison (however separately), i.e., both performed measurements at the same time, placed specimens into the oven, removed them from the oven, cooled and measured them again.

Accuracy and time factors were essential in this test since, except for the human factor, all other conceivable variables were removed from this test.

6.2 Procedural Differences

The sole variation in operator procedure was in regard to the way each gaged the specimen. This was done twice by New Jersey i.e., the specimen was gaged then turned over and regaged, whereas DuPont gaged the specimen only once.

6.3 Equipment Differences

While NJDOT used only 4x6x7/16 inch plates with four threaded tie bars, DuPont had two sets of plates, one set 5x7x1/2 inch with four threaded tie bars and the other 3x6x1/2 inch with two threaded bars to compress said plates as shown in Figures 4 and 5.

Chrome plated plates were used by New Jersey Department of Transportation whereas DuPont used steel plates and a thin chrome plated material.

The arrangement of the plates and specimens differed between New Jersey Department of Transportation and DuPont (Figures 4 and 5). However, plate bending calculations for each set of plates indicated that plate deflections would not introduce a measurable difference in results.

The DuPont dial micrometer foot was 0.25 inch in diameter compared to the NJDOT dial micrometer foot diameter of 1.00 inch. Since only DuPont's micrometer was used this difference could not have any effect in this test. Nevertheless, when tests were performed with both micrometers, the maximum difference in gage readings was 0.003 inch which could only affect the compression set test results by a maximum of 2%.

All these differences can be considered to be of a minor nature and were not expected to influence the outcome of the tests.

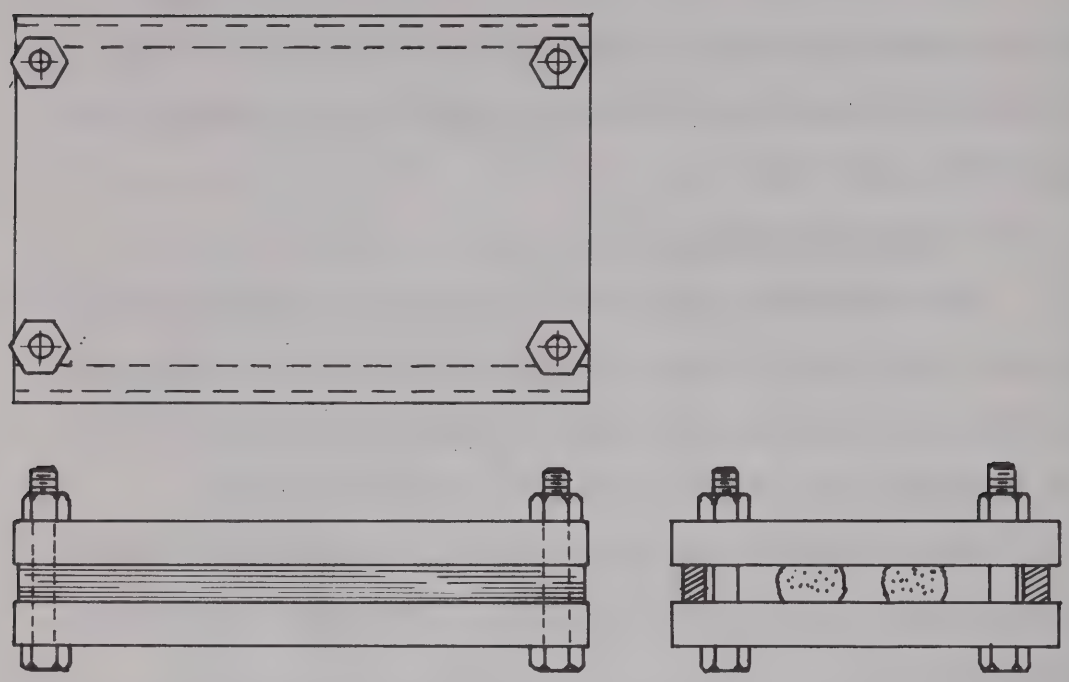

FIG. 4 - FOUR THREADED TIE-BARS DEVICE FOR COMPRESSION SET TEST - METHOD "B".

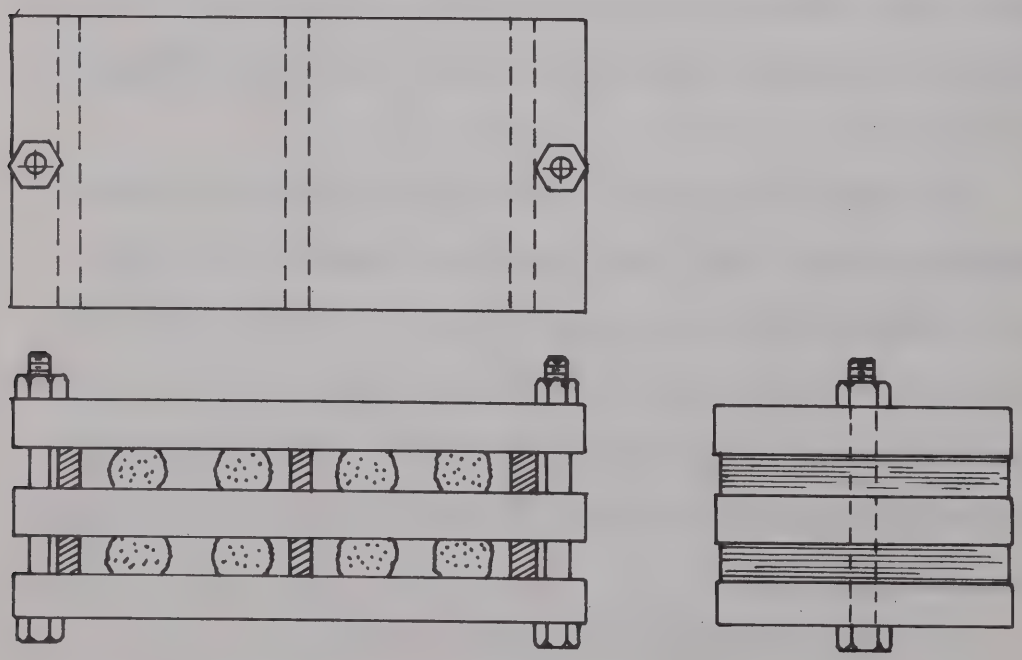

FIG. 5 - TWO THREADED TIE-BARS DEVICE FOR COMPRESSION SET TEST METHOD "B".

6.4 Results and Analysis

The scheduling of the tests and a summary of the results are presented in Table 11. Although it was originally intended to use only Type III specimens, a quantity of Type I specimens had been prepared by DuPont so these were included in the test also. However, for this reason it was no longer possible to provide the originally intended 30 specimens for each operator and each material as planned. Because all tests could not be performed at the same time, it seemed advisable to consider batches of specimens tested on different days as separate tests. A loss of specimens due to slippage further reduced the number of specimens made available for comparison (see Table 11).

Nevertheless, from the data in Table 11 it appears that there is a significant difference between some of the results obtained by the two operators. However, unlike the previous comparison of DuPont and New Jersey tests, the difference does not take the form of a consistent bias. Of the six comparisons that were made, DuPont had the higher value three times and New Jersey had the higher value an equal number of times. The differences ranged from nearly zero up to about 5 units compared to a nearly constant difference of about 10 units in the earlier tests.

The occurrence of statistically significant differences with either operator likely to have the higher value suggests that there apparently is some other component of variability present in these tests which is not being accounted for. It probably is not related to the known procedural and equipment differences already mentioned

TABLE 11 SUMMARY OF COMPARATIVE COMPRESSION SET TEST RESULTS

Date Tested	Type	Lot	Operator	Number of Original Specimens	Number of Completed Specimens*	Mean Compression Set (%)	Standard Deviation	Are Means Significantly Diff. at 95% Conf. Level?
7-27-76 to 7-30-76	I	68	DuPont	16	16	28.65	0.69	Yes
			New Jersey	15	15	27.96	0.52	
		69	DuPont	30	30	34.46	0.69	Yes
			New Jersey	30	30	31.07	2.04	
7-27-76 to 7-30-76	III	68	DuPont	15	10	33.92	0.70	Yes
			New Jersey	15	10	38.64	1.11	
		69	DuPont	15	13	44.42	2.02	No
			New Jersey	15	3	47.06	2.60	
7-30-76 to 8-2-76	III	68	DuPont	15	15	34.47	1.87	No
			New Jersey	15	15	34.95	1.30	
		69	DuPont	15	15	43.93	2.32	Yes
			New Jersey	15	15	39.94	1.58	

*Some specimens became unusable due to slippage between plies during the test.

because these remained essentially constant and if they had any effect it would be expected to show up as a consistent bias. A possible additional source of variability could have been the material itself although the pellets were selected in a random manner which should have precluded this. It could be that the temperature gradient in the oven was not entirely uniform and that this might have affected the results. However, this is considered unlikely because of the type of oven and steps taken to prevent such an unusual occurrence.

The mere existence of a significant difference in test results with no apparent significant differences in material, equipment, or procedure suggests a weakness of the test method itself. It appears that the test method as prescribed by ASTM D-395-69 has imprecisions and/or operations that invariably lead to widely variable results. One step that has been shown to be critical is the thickness measurement used in calculations of compression set.

In the formula $C = [t_o - t_i)/(t_o - t_s)] \times 100$ the minute variation in measurement of original or final thickness of specimen results in considerable variability of compression set percentage points. For example if in a 0.5 inch, four ply specimen a gap of only 0.003 inch would exist between each ply due to some irregularity, an erroneous original thickness of 0.509 inch would be measured. Having in the compression set test the final thickness of say 0.475 inch would result in the computational variation of +6%. In preparation of specimens it is impossible to avoid at times buckling or bulging of the individual plies which in the process of plying them into specimens can provide enough variability to cause this phenomenom.

It appears that the solution to this probem rests with the rubber specialists and the appropriate ASTM Committees. It is apparent that the compression set test is not an extremely precise test and that caution should be exercised in its use. However, until better methods for testing this material (rubber) are available there is no alternative but to use the test in the prescribed manner. The results obtained must then be treated in light of imprecision associated with this test.

7. Comparison of Tests on Sealer Webbing and Shell Material

Before proceeding to the development of compression set acceptance limits for the New Jersey laboratory there is another important comparison that must be made. This involves a comparison of samples cut from different parts of the actual sealer, i.e., from the outer shell or from the inner webbing. Type III specimens from Lots 18, 25, 71, 406, and 474 were used for this comparison and the results are listed in Table 12. It is apparent even without statistical analysis that there is less variability associated with the tests on the "shell" material since these standard deviations are substantially lower. This may be due to a difference in curing conditions between the outer and inner portions of an extruded sealer or it might be caused by a greater geometric variability of the specimens constructed from "inner" material. Regardless of the cause, it suggests that more precise tests could be made by using "outer" material only, provided that "outer" material is representative of the true quality of the lot. However, when this condition was checked a serious problem was discovered. If either outer or

inner material alone was used, one of the known "unsatisfactory" lots scores better (lower) than one of the known "satisfactory" lots. However, when both outer and inner material are combined, all the satisfactory lots score lower than any of the unsatisfactory lots as desired. Therefore, the subsequent analysis of tests with known satisfactory and unsatisfactory material will utilize both inner and outer material results.

8. Development of Compression Set Acceptance Limits

In Table 12, using the data for webbing and shell material combined, the worst "satisfactory" lot has a mean compression set of 31.6 while the best "unsatisfactory" lot has an average value of 32.4. It would seem logical then to select an acceptance limit between these two values and make the sample size large enough to easily discriminate between satisfactory and unsatisfactory material. This requires the construction of a series of operating characteristic curves and in order to do this it is first necessary to known the value of the standard deviation for all possible compression set levels.

Figure 6 shows the approximate relationship between standard deviation and average compression set for six typical lots which are being evaluated. Since it was not considered practical to perform a formal regression analysis on this small amount of data, the curve was drawn by eye through the six points shown in Figure 6. It was reasoned that the curve should pass very nearly through the origin since, if there actually were no compression set, there would be very little variability in the measurements. A mathematical expression was then derived which closely approximated this curve. It

TABLE 12 COMPARISON OF TEST RESULTS ON

INNER WEBBING AND OUTER SHELL MATERIAL

Lot No.	Quality	Inner Webbing			Outer Shell			Webbing and Shell Material Combined		
		Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)	Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)	Number of Tests (N)	Mean Comp. Set ($\bar{X}$)	Standard Deviation (S)
406	Satisfactory	22	27.2	5.34	44	27.0	2.11	66	27.1	3.49
474	Satisfactory	66	30.6	2.42	70	32.5	2.20	136	31.6	2.49
6	Unsatisfactory	19	42.3	4.87	49	45.8	5.77	68	44.8	5.72
18	Unsatisfactory	32	27.7	4.50	52	37.8	3.03	84	34.0	6.13
25	Unsatisfactory	15	33.9	4.38	14	30.8	1.99	29	32.4	3.73
71	Unsatisfactory	27	39.2	6.26	29	48.3	4.12	56	43.9	6.94

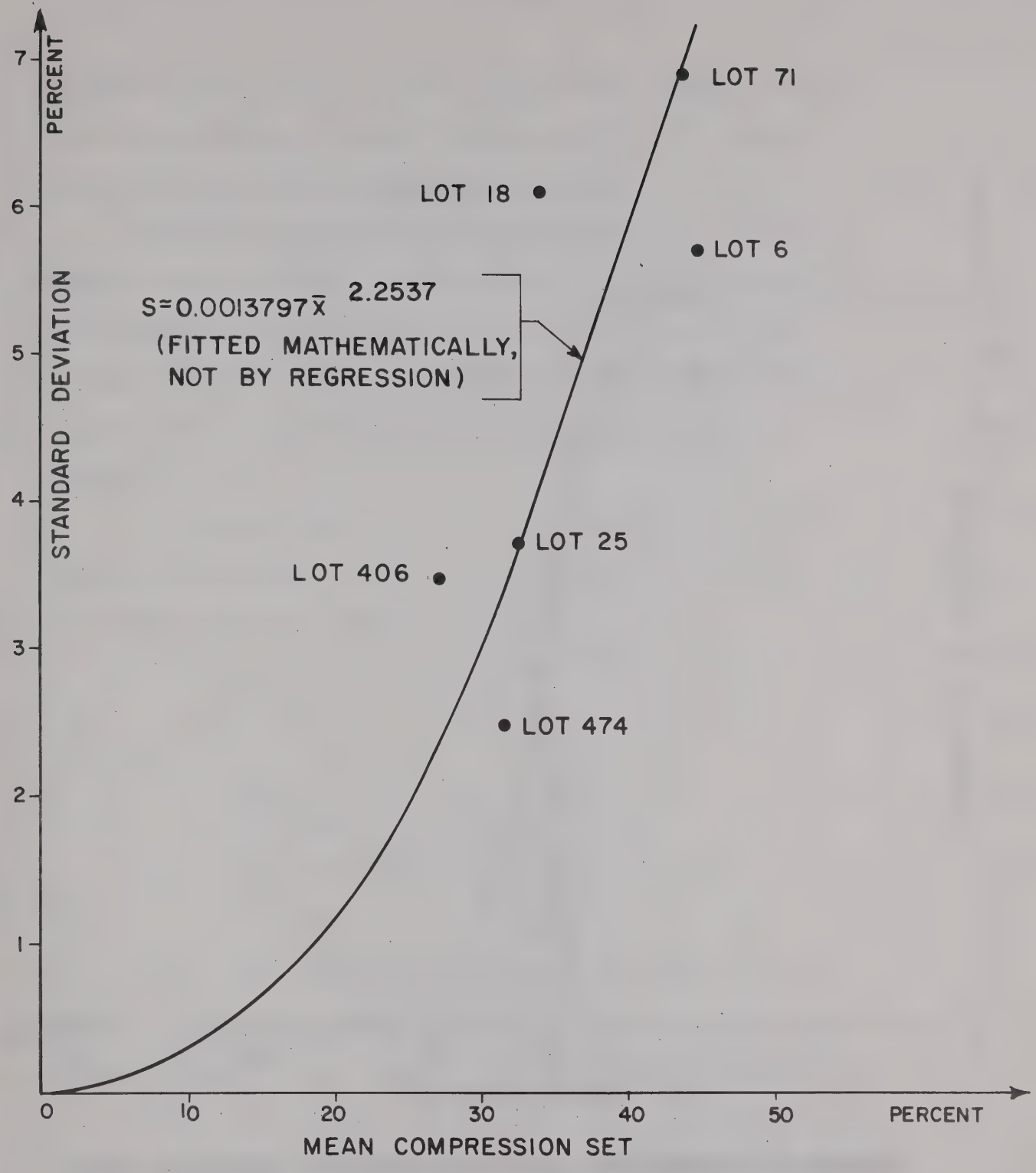

FIGURE 6. APPROXIMATE RELATIONSHIP OF STANDARD DEVIATION AND MEAN COMPRESSION SET

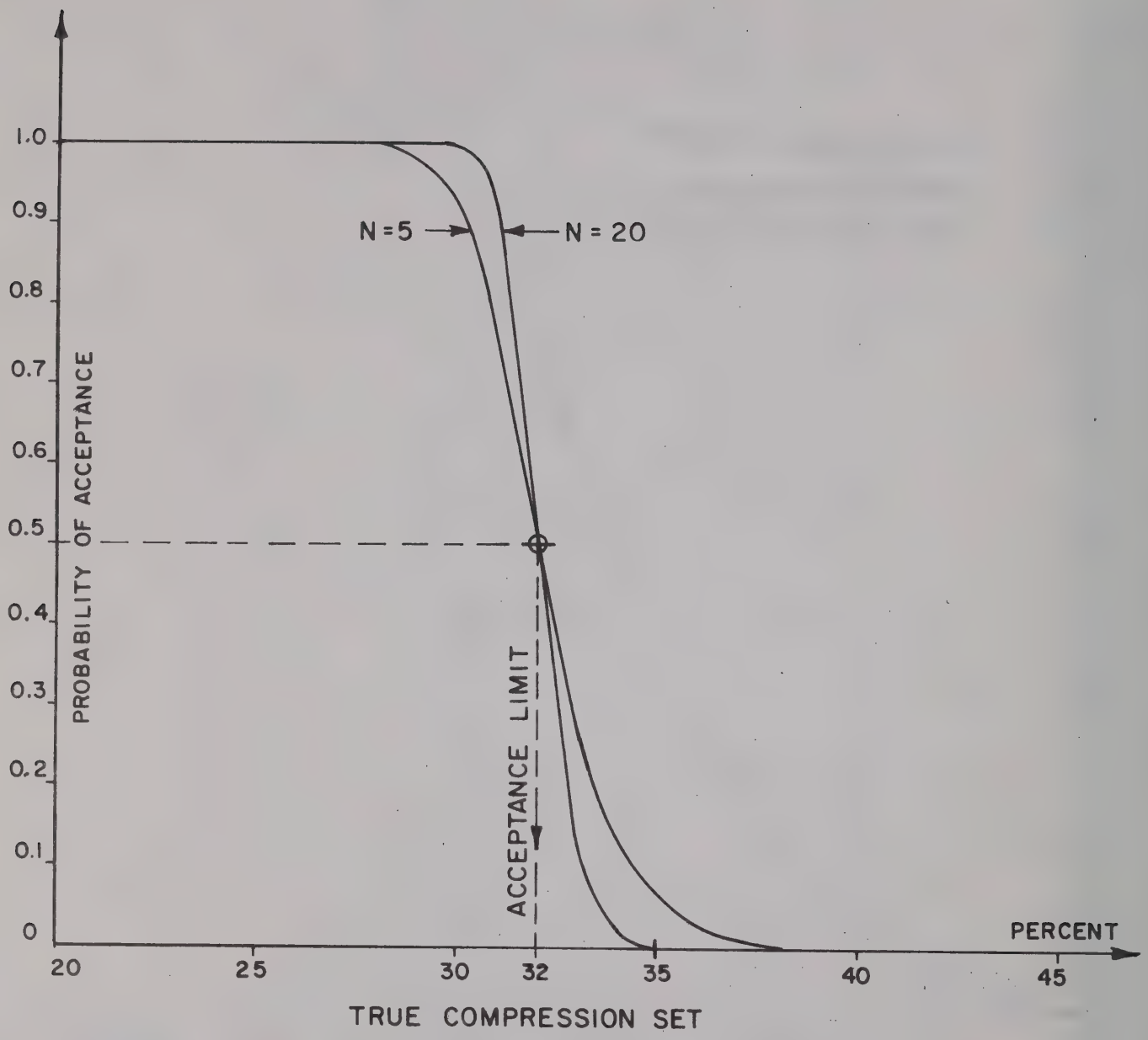

FIGURE 7. OPERATING CHARACTERISTIC CURVES FOR COMPRESSION SET TEST WITH AN ACCEPTANCE LIMIT OF 32 % (VARIABILITY ASSUMED KNOWN).

is believed that this will produce more realistic results than the alternative of using a constant (pooled) value of the standard deviation in developing the operating characteristic curves.

In order to develop an appropriate acceptance limit which yields reasonable producer's and consumer's risks, the values of the standard deviation obtained from Figure 6 will be assumed to be known exactly. Once this limit has been chosen, an acceptance procedure will be developed which does not rely upon this assumption.

For a trial acceptance limit of 32%, points on the operating characteristic curves in Figure 7 are obtained by first determining the normal variate "z" from the expression

$$z = \frac{32-\bar{X}}{\sigma/\sqrt{N}}$$

in which

$\bar{X}$ = mean compression set of sample

σ = std. dev. (from Figure 5)

N = sample size

Then, using a table of the standard normal distribution, the probability of acceptance associated with each "z" value is found and plotted in Figure 7. For clarity, only the curves for sample sizes N = 5 and N = 20 are shown in this figure. For a particular sample size (5 or 20) and a particular value of compression set, the probability of acceptance of the material can be determined from these curves.

To provide a clearer indication of how this specification would perform, the probability of acceptance of the six lots used to develop this specification is listed in Table 13 for sample sizes ranging from 5 to 100. Although a sample size of 100 may not be practical, the results tend to indicate limiting values for the probabilities of acceptance and rejection. If a sample size of 10 is chosen, for example, the probabilities of acceptance for the two "satisfactory" lots are 1.00 and 0.64. If these two lots can

TABLE 13 PERFORMANCE OF COMPRESSION SET ACCEPTANCE PROCEDURE WITH AN ACCEPTANCE LIMIT OF 32%

Lot No.	Quality	Mean Comp. Set ($\bar{X}$)	Probability of Acceptance for Sample Size (N)			
			N = 5	N = 10	N = 20	N = 100
406	Satisfactory	27.1	1.00	1.00	1.00	1.00
474	Satisfactory	31.6	0.60	0.64	0.70	0.88
6	Unsatisfactory	44.8	0.00	0.00	0.00	0.00
18	Unsatisfactory	34.0	0.14	0.07	0.02	0.00
25	Unsatisfactory	32.4	0.39	0.37	0.32	0.14
71	Unsatisfactory	43.9	0.00	0.00	0.00	0.00

be thought of as typical of "satisfactory" lots that might be tested in the future, then this specification would (on the average) accept satisfactory material about 80% of the time. And if the remaining three lots are representative of "unsatisfactory" lots, the specification will accept them only about 15% of the time. In terms of producer's and consumer's risks these risks are 20% and 15%, respectively which is rather large for both. Even with a sample size N = 20, they are still fairly large at 15% and 11%, respectively. However, if no other satisfactory testa are available, it may be necessary to tolerate risks of this magnitude if an acceptance procedure based on compression set tests is desired. An alternative, of course, would be to change the acceptance limit to some value other than 32, but for a given sample size any lessening of one risk will be offset by an increase in the other risk so little could be accomplished in this manner.

8.1 Proposed Acceptance Procedure

It should be noted that the mathematical expression for the standard deviation from Figure 6 is, in effect, a precision statement and implies that the combined variability of the product and the measurement technique is well established. Although the predicted values might be quite accurate on the average, this would be a risky assumption for any particular case. Since we have already seen that an acceptance procedure based on compression set will require a fairly large sample size (on the order of 20), it will be more appropriate to estimate the variability from each sample. The acceptance procedure developed in this manner will utilize a t-statistic to judge the acceptability of the product. If the average compression

set is statistically significantly greater than 32.0, the material would be rejected; otherwise it would be accepted. The steps in this procedure are as follows:

1. Select a random sample of size n = 20, perform the necessary tests, and record the results.

2. Calculate the mean ($\bar{X}$) and standard deviation (S) of the test results:

$$\bar{X} = \frac{\Sigma X}{n} ;$$

$$S = \sqrt{\frac{n\Sigma X^2 - (\Sigma X)^2}{n(n-1)}} ;$$

3. Calculate $t = \frac{\bar{X} - 32.0}{S/\sqrt{n}}$;

4. If $t > 1.729$ (95% confidence level), reject the material; otherwise accept.

Note: If $\bar{X} \leq 32.0$, accept the material; there is no need to calculate the t value.

9. Summary and Conclusions

Phase II of this research has provided unique information concerning the relationship of material specifications to a sealers field performance. In the controlled performance test under true field exposure conditions, out of 16 sealers, 25% have shown exceptionally poor performance. Nevertheless, all of these sealers have passed the current material specification equally well. This obviously prompted further investigation into possible causes and remedies. Since the subject research has indicated that this is strictly a problem associated with the manufacturing of a product, assistance of a rubber manufacturer, the DuPont deNemours organization,

was sought and received. As a result of DuPont laboratory investigations, no specific causes for the poor performance of certain of the sealers could be established. However, a prolonged Compression Set test and High Temperature Recovery test were proposed as possible screening tests in an attempt to further refine the subject material specification. In the subsequent joint efforts the High Temperature Recovery test, performed on 6 inch specimens under 50% compression and aged for 210 hours at 212°F was proven to be impractical.

The Compression Set test, on the other hand, performed as per ASTM D-396-69 Method B (2) modified by Type III plied pellets, appeared to be best suited for the purpose. To ascertain the best possible results various bipartisan testing efforts were carried out in both DuPont's and the Department's laboratories.

The Type III plied pellets (0.51 $\pm$ 0.01 inch diameter and 0.50 $\pm$ 0.02 inch thick) were found to produce consistent results.

While comparison between laboratories showed consistently differing results, no fault in procedures or equipment or personnel could be established in either organization. The comparison performed in the DuPont laboratory between DuPont and NJDOT operators produced equally higher values for each operator indicating some unidentifiable components of variability in these tests. In fact, comparison tests between New Jersey operators in the New Jersey laboratory produced no sufficiently significant results. In general, the joint evaluative efforts involving the Compression Set test tended to show the test to be relatively imprecise. Unfortunately, without a viable alternate, it appears necessary to rely upon this test.

The statistical analysis indicates probably the most reasonable "acceptance limit" for the Compression Set test as being 32% with a sample size of 20 pellets divided equally between sealer's shell and webbing. In terms of producer's and consumer's risks, use of this value unfortunately yields fairly large risks of approximately 15% and 11%. However, since current specifications cannot at all isolate certain basic inconsistencies in the production of sealer material, and since there apparently are no other satisfactory tests available at this time, there seem to be no other alternatives. This test provides at least some protection against accepting defective material.

As previously indicated, the solution to the problem is really outside the realm and capabilities of the consumer's research especially since the production of this material is so shrouded in secrecy by every manufacturer. Considering the results of DuPont's materials tests and those from our comparison of methods of curing and formulations of material the influence of the manufacturing processes on the functional characteristics of the product (the sealers) is clearly evident. Indeed, it appears that the solution of this problem rests with the rubber specialists and the appropriate ASTM Committees.

10. Recommendations

The recommendations that can be made based on the findings of this research effort are as follows:

1. The New Jersey Department of Transportation should adopt the Compression Set Test as part of the standard specification for Preformed Elastomeric Compression Joint Sealers. The test should be performed in accordance with ASTM D-395-69 Method B specifications

modified by Type III plied pellets. For tests conducted in the Department of Transportation's Trenton laboratory the acceptance limit should be @32% with a sample size of not less than 20 pellets. The acceptance procedure developed in this manner will utilize a t-statistic to judge the acceptability of the product. If the average compression set is statistically significantly greater than 32%, the material would be rejected; otherwise it would be accepted. A one year period of applying the test without the acceptance limit should be considered as a means of gaining experience in interpreting the test results and to obtain data on failure rates to be expected. The failure rate information might be useful in influencing sealer manufacturers to upgrade their manufacturing processes.

2. To further minimize the producer and consumer risks associated with the Compression Set test, manufacturers of this product are urged to make every effort to make this basically excellent and very needed product uniformly good. This research has shown that a good quality product is quite within existing sealer fabrication limits.

3. The manufacturers are further urged to develop material qualification tests in order to assure production of uniformly good material.

4. Consumers, however, are no less strongly requested to make efforts to better comprehend problems brought forth herein, in order to encourage manufacturers in their efforts to provide better products.

5. Investigation utilizing the model joint machines and test methods of this research can be extended to achieve results suggested in the above recommendations.

11. References

1. New Jersey Department of Transportation, Division of Research and Evaluation, Bureau of Structures and Materials, "Preformed Elastomeric Joint Sealer in Bridge Construction", Report #29.

2. ASTM Standards, Part 38: D-395-69, Method B, Modified, Test for Compression Set of Vulcanized Rubber.

3. New Jersey Department of Transportation's Standard Specification for Preformed Elastomeric Compression Joint Sealers, Article 904.04.

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